Attachment D

U.S. EPA ERT modified NIOSH Method 6009

CONTENTS

CONTENTS (Cont'd)

* This section affected by Revision 2.0

SUPERCEDES: SOP #1827; Revision 1.0; 01/16/96.

1.0 SCOPE AND APPLICATION

This method is a modification of NIOSH Method 6009 for mercury (Appendix A). It is applicable to the analysis of indoor air samples of volatilized elemental mercury (Hg) collected on solid sorbent material (typically Hopcalite) contained in glass collection tubes. The sorbent sample is digested and the Hg concentration is determined by the cold-vapor Atomic Absorption (AA) spectroscopy technique. The method is simple, rapid, and relatively free of matrix interferences.

Detection limits, sensitivity, and optimum ranges for Hg analysis will vary with the sorbent material, volume of air sampled, and models of atomic absorption spectrophotometers used.

This standard operating procedure may be varied or changed as required, depending upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed will be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 SUMMARY OF METHOD

Indoor air samples of elemental Hg are collected on solid Hopcalite sorbent material contained in glass tubes. Air is pumped through the sorbent with a personal sampling pump which can be programmed for collection time and **flow rate [typically 0.25 to 0.5 liters per minute (L/min)]**. Pump flow rate is initially calibrated against a rotameter reference and is checked before/after sample collection. Sampling stations are typically set up in several locations within the residence with duplicate stations in areas suspected of high level contamination. Refer to manufacturer's instructions for proper setup and operation of sampling equipment and ERT/REAC SOP #2119, *Air Sampling for Metals*.

The sorbent material from the collection tube (typically 200 mg in a single section) is quantitatively transferred to a 100-milliliter (mL) volumetric flask. The sample is digested by first adding 2.5 mL of concentrated nitric acid followed by 2.5 mL of concentrated hydrochloric acid. After digestion is complete, the sample is diluted to volume with deionized water.

The sample is analyzed by the cold-vapor AA spectroscopy technique with no additional dilutions. The principle is essentially the same as direct aspiration AA, except a cold-vapor generator system, rather than a flame, is used to atomize the sample. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The intensity of the transmitted radiation decreases in proportion to the amount of the ground-state element in the vapor. A monochromator isolates the characteristic radiation from the hollow cathode lamp and a photosensitive device measures the attenuated transmitted radiation.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Sample holding times, suggested collected volume, preservative, and type of containers are as follows:

Holding Measurement	Collection Volume Req. (L)	Type of Containers	Preservative	Time
Mercury in air:				
Solid Hopcalite sorbent	10 - 200 L [1]	sorbent tube	sorbent tube, 25° C	30 days

[1] The volume of air collected is directly related to detection limit; the larger the volume, the lower the detection limit.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Although the method minimizes dilution and sample matrix effects, the technique is not completely interference free. Inorganic and organic Hg compounds may cause a positive interference

Cross-contamination and contamination of the sample can be major sources of error because of the sensitivities achieved with the cold-vapor AA spectroscopy technique. The sample preparation work area should be kept scrupulously clean. All glassware should be cleaned as directed in Section 5.3. Special attention should be given to reagent blanks in both analysis and in the correction of analytical results.

5.0 EQUIPMENT/APPARATUS

5.1 Atomic Absorption Spectrophotometer

A single- or dual-channel, single- or double-beam instrument having a grating monochromator, Hg hollow cathode lamp, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nanometers (nm), and provisions for interfacing with a strip-chart recorder or computer, printer, autosampler, and Hg cold-vapor generation system.

5.2 Strip-Chart Recorder or Integrator

A recorder is useful to provide a permanent record and for easy recognition of any problems with the analysis.

5.3 Glassware

All glassware, polypropylene, or Teflon™ containers, including sample bottles, should be washed in the following sequence: detergent, tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water, and Type I water. If it can be documented through an active analytical quality control program, using spiked samples and reagent blanks, that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

6.0 REAGENTS

All reagents and standard solutions are prepared and documented in accordance with ERT/REAC SOP #1012, *Preparation of Standard Solutions*.

6.1 Type I Water (ASTM D1193)

Use Type I water for the preparation of all reagents and calibration standards and as dilution water.

6.2 Concentrated Nitric Acid (HNO_3)

Use commercially available high-purity, spectrograde acid certified for AA use.

6.3 Concentrated Hydrochloric Acid (HCl)

Use commercially available high-purity, spectrograde acid certified for AA use.

6.4 Stock Mercury Solutions

Use a commercially available Hg standard solution or prepare a 1000 micrograms per milliliter (: g/mL) stock standard solution from high purity mercuric oxide (HgO) using Type I water and redistilled HCl. Dissolve 1.0798 grams (g) of dry HgO in 50 mL of 1:1 HCl, then dilute to one liter (1 L) with Type I water.

6.5 Stannous Chloride, 10% in 1:1 HCl

Dissolve 20 grams (g) reagent grade stannous chloride in 100 mL concentrated HCl. Slowly add this solution to 100 mL Type I water and mix well. Prepare fresh daily or each time calibration standards are prepared.

6.6 Calibration Standards

For those instruments which do not read out directly in concentration, a calibration curve is prepared to cover the appropriate concentration range. For best results, intermediate and working standards should be prepared fresh each time a batch of samples is analyzed. A blank and a minimum of three working standards in the linear part of the calibration range should be used to calibrate the AA instrument.

Prepare all calibration and all check standards using the procedures outlined in Section 7.0. All QC solutions are prepared by spiking blank sorbent media. This matches the sample matrix and, thereby, minimizes sample matrix effects.

7.0 PROCEDURE

7.1 Sample Preparation

1. Quantitatively transfer the Hopcalite sorbent and the front glass wool plug from each sampler tube into a 100-mL volumetric flask.

2. Add 2.5 mL of concentrated HNO_3 followed by 2.5 mL concentrated HCl.

   NOTE: The Hg must be in the oxidized state to avoid loss. For this reason, the nitric acid must be added first.

3. Allow the sample to stand for 1 hour or until the black Hopcalite sorbent is dissolved. The solution will turn dark brown and may contain undissolved material.

4. Carefully dilute to 100 mL with Type I water.

5. The final sample solution (blue to blue-green in color) contains 2.5 percent HNO_3, 2.5 percent HCl, and is analyzed for Hg with no additional dilution (except for samples containing high concentrations of Hg).

7.2 Calibration Standards

Prepare a blank, and a minimum of three working standards in graduated amounts in the linear part of the calibration range (0.2 to 10.0 : g/L) by spiking blank sorbent media (from unused Hopcalite tubes) with known amounts of Hg. Dissolve the blank sorbent media, using steps 1 - 3 of the procedure outlined in Section 7.1. Spike each standard solution with the appropriate amount of Hg, and dilute to volume per step 4 of the procedure outlined in Section 7.1.

7.3 Method Blank

Prepare the method blank (reagent blank) from blank sorbent media. Prepare the method blank using the procedure outlined in Section 7.1 at the same time the samples are prepared. A minimum of one method blank per batch of 20 samples or less must be prepared.

7.4 Laboratory Control Standard

Prepare the laboratory control standard (LCS) by spiking blank sorbent media with a known amount of Hg (at or near midrange of the calibration curve). Use an independent source of Hg (different than that used to prepare calibration standards) for the LCS sample. Prepare the LCS sample using the procedure outlined in Section 7.1 at the same time the samples are prepared.

7.5 Calibration Verification Standards

Prepare initial calibration verification (ICV) and continuing calibration verification (CCV) standards by spiking blank sorbent media with known amounts of Hg (at or near midrange of the calibration curve). Prepare ICV and CCV standards using the procedure outlined in Section 7.1.

7.6 Method Detection Limit Standard

Prepare the method detection limit (MDL) standard by spiking blank sorbent media with a known amount of Hg at the expected MDL (typically half the concentration of the lowest calibration standard). Prepare the MDL standard using the procedure outlined in Section 7.1.

7.7 Blank Spike/Blank Spike Duplicate

Prepare blank spike (BS) and blank spike duplicate (BSD) samples by spiking blank sorbent media with known amounts of Hg (5 to 10 times the detection limit). Spike blank sorbent media with appropriate amounts of Hg and prepare BS/BSD samples using the procedure outlined in Section 7.1. BS/BSD samples are prepared in the laboratory to monitor precision and accuracy of the method.

Use the BS/BSD samples delivered to the laboratory with unknown samples, or blank tubes from the same lot if none are provided with the samples.

7.8 Lot Blank

One unopened sampling tube must be prepared and analyzed as a lot blank with each sample lot of twenty tubes or less per project. The lot blank tube is delivered to the laboratory with the samples, broken up without disturbing the contents, and then prepared using the procedure outlined in Section 7.1.

7.9 Trip Blank

One blank tube from the same lot is included as the trip blank. The tube is broken and carried to the site in the same type of container as the samples. It is delivered to the laboratory with the samples and is prepared using the procedure outlined in Section 7.1.

7.10 Field Blanks

One or more blank tubes from the same lot are included as field blanks. The tube is broken and carried to the sampling stations at the site but no air is sampled. Field blanks are delivered to the laboratory with the samples and are prepared using the procedure outlined in Section 7.1.

7.11 AA Calibration and Measurement

Differences between the various makes and models of satisfactory AA instruments and cold-vapor generators prevent the formulation of detailed instructions applicable to each system. The analyst should follow the manufacturer's operating instructions for a particular instrument and cold-vapor generator system.

Analyze the working standards together with the samples and blanks. Analyze the full set of working standards at the beginning of the run to establish the initial calibration curve. Analyze additional standards during sample analysis to confirm instrument response (see Section 9.0).

Samples with a high concentration of Hg should be diluted into the linear calibration range (see Section 9.6).

8.0 CALCULATIONS

For determination of concentration, read the Hg value (B) in µg/L from the calibration curve or directly from the read-out system of the instrument. Calculate the concentration of Hg in the sample aliquot (A) as follows:

$$A = \mu g/L \text{ Hg in sample aliquot} = B\,\frac{D + C}{D}$$

where:

B	=	Concentration of Hg from the calibration curve (µg/L)
C	=	Amount of acid blank matrix used for dilution (mL)
D	=	Sample aliquot used for dilution (mL)

Note: If no dilution was required, C = 0, and A = B.

Calculate the amount of Hg (W) for each Hopcalite tube:

$$W = \mu g/tube\ Hg = A\ (0.1)$$

where:

A	=	Concentration of Hg in the sample aliquot (:g/L)
0.1	=	Final solution volume (L/tube)

Calculate the concentration (:g/m^3) of Hg in the air volume sampled:

$$\mu g/m^3\ Hg = 1000\ \frac{W - BLK}{V}$$

where:

W	=	Amount of Hg in each Hopcalite tube (:g/tube)
BLK	=	Amount of Hg present in the method blanks (:g/tube)
V	=	Volume of air sampled (L/tube)

NOTE: Report :g/tube results for lot blank, trip blank, field blank(s), and BS/BSD samples because no air volume is collected for these samples.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

All quality control data should be documented and available for reference or inspection.

9.1 Initial Calibration/Calibration Check

A calibration curve must be prepared each day with a minimum of a blank and three standards (Sections 7.2 and 7.11). The initial calibration must be verified by analysis of the method blank (Section 7.3), the ICV standard (Section 7.5), and the LCS sample (Section 7.4). The ICV and LCS results must be within $\pm$ 10 percent of the true value.

9.2 Method Detection Limit

The MDL standard (Section 7.6) must be analyzed at the beginning and end of sample analysis to verify the Hg MDL. MDL standard results must be within $\pm$ 20 percent of the true value. The MDL is 0.10 :g/L (0.05 :g/m^3 for a 200-L air sample) using a Varian Spectra AA-300™ equipped with a VGA-76 vapor generator accessory.

9.3 Continuing Calibration Verification

If 20 or more samples per day are analyzed, the working standard curve must be verified by analyzing the method blank and the CCV standard after every 10 samples. CCV results must be within $\pm$ 10 percent of the true value.

9.4 Blanks

One lot blank, one trip blank, and a minimum of one field blank must be prepared and analyzed with each sample lot.

9.5 BS/BSD Samples

At least one BS and one BSD sample should be analyzed after every 10 samples or with each sample lot to verify precision and accuracy of the method.

BS/BSD percent recovery (%R) should be within 75-125 percent (advisory only) and calculated as:

$$\%R = \frac{(R - BLK)}{SA} \times 100$$

where:

R	=	BS or BSD sample result (:g/tube)
BLK	=	Amount of Hg present in the lot blank (:g/tube)
SA	=	Spike added (:g/tube)

The Relative Percent Difference (RPD) for duplicate samples should be within 80-120 percent (advisory only) and calculated as:

$$RPD = \frac{(S - D)}{(S + D)/2} \times 100$$

where:

S	=	BS sample result
D	=	BSD sample result

9.6 Dilution Analysis

If the Hg concentration of any sample exceeds the initial calibration range, that sample must be diluted and reanalyzed. Use the results of the original analysis to determine the approximate dilution factor required to get a Hg concentration within the upper half of the initial linear calibration range. Analyze the diluted sample aliquot. Adjust the dilution factor if the Hg concentration is not within the desired range. Report results for the diluted aliquot with the smallest dilution factor which produces a Hg concentration in the upper half of the linear calibration range (see Section 8.0).

10.0 DATA VALIDATION

Data is considered valid if all the procedures for analyzing Hg in air are followed and QA/QC criteria are met.

11.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. The laboratory is responsible for following the chemical hygiene plan and laboratory safety program regarding the safe handling of the chemicals specified in this method.

When working with potentially hazardous materials, refer to U.S. EPA, Occupational Safety and Health Administration (OSHA) and corporate health and safety practices. More specifically, refer to ERT/REAC SOP #3013, *REAC Laboratory Safety Program*.

12.0 REFERENCES

Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency. September, 1986. *Test Methods for Evaluating Solid Waste.* Third Edition. SW-846.

NIOSH Manual of Analytical Methods, Method 6009, Mercury. August 15, 1994. Issue 2.

ERT/REAC SOP #1818, *Determination of Metals by Atomic Absorption (AA) Methods*.

ERT/REAC SOP #2119, *Air Sampling for Metals, [NIOSH METHOD 7300, Elements]*

FORMULA: Hg

M.W.: 200.59

MERCURY
METHOD: 6009
ISSUED: 5/15/89

OSHA: 0.05 mg/m^3 (skin)
NIOSH: 0.05 mg/m^3 (skin) [1]
ACGIH: 0.05 mg/m^3

PROPERTIES: liquid; d 13.55 g/mL @ 20 °C; BP 356 °C; MP -39 °C; VP 0.16 Pa (0.0012 mm Hg; 13.2 mg/m^3) @ 20 °C

SYNONYMS: quicksilver; CAS# 7439-97-6.

SAMPLING

SAMPLER: SOLID SORBENT TUBE
(Hydrar in single section, 200 mg)

FLOW RATE: 0.15 to 0.25 L/min

VOL-MIN: 2 L @ 0.05 mg/m^3
-MAX: 100 L

SHIPMENT: routine

SAMPLE STABILITY: 30 days @ 25 °C [2]

FIELD BLANKS: 10% of samples
MEDIA BLANKS: at least 3 per set

ACCURACY

RANGE STUDIED: 0.002 to 0.8 mg/m^3 [3]
(10-L samples)

BIAS: not significant [2,3]

OVERALL PRECISION (s_r): not determined

MEASUREMENT

TECHNIQUE: ATOMIC ABSORPTION, COLD VAPOR

ANALYTE: elemental mercury

DESORPTION: conc. HNO_3/HCl @ 25 °C, dilute to 50 mL

WAVELENGTH: 253.7 nm

CALIBRATION: standard solutions of Hg^{++} in 1% HNO_3

RANGE: 0.1 to 1.2 µg per sample

ESTIMATED LOD: 0.03 µg per sample

PRECISION (s_r): 0.042 @ 0.9 to 3 µg per sample [4]

APPLICABILITY: The working range is 0.01 to 0.5 mg/m^3 for a 10-L air sample. The sorbent material irreversibly collects elemental mercury. A prefilter can be used to exclude particulate mercury species from the sample. The prefilter can be analyzed by similar methodology. The method has been used in numerous field surveys [4].

INTERFERENCES: Inorganic and organic mercury compounds may cause a positive interference. Oxidizing gases, including chlorine, do not interfere.

OTHER METHODS: This replaces method 6000 and its predecessors, which required a specialized desorption apparatus [5,6,7]. This method is based on the method of Rathje and Marcero [8] and is similar to the OSHA method ID 145H [3].

MERCURY METHOD: 6009

REAGENTS:

1. Water, organics-free, deionized.
2. Hydrochloric acid (HCl), conc.
3. Nitric acid (HNO_3), conc.
4. Mercuric oxide, reagent grade, dry.
5. Calibration stock solution, Hg++, 1000 µg/mL. Commercially available or dissolve 1.0798 g of dry mercuric oxide (HgO) in 50 mL of 1:1 hydrochloricacid, then dilute to 1 L with deionized water.
6. Intermediate mercury standard, 1 µg/mL. Place 0.1 mL 1000 µg/mL stock into a 100 mL volumetric containing 10 mL deionized water and 1 mL hydrochloric acid. Dilute to volume with deionized water. Prepare fresh daily.
7. Stannous chloride, reagent grade, 10% in 1:1 HCl. Dissolve 20 g stannous chloride in 100 mL conc. HCl. Slowly add this solution to 100 mL deionized water and mix well. Prepare fresh daily.
8. Nitric acid, 1% (w/v).

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame sealed ends with plastic caps, containing one section of 200 mg Hydrar held in place by glass wool plugs (commercially available from SKC, Inc., Cat. #226-17-1).
   NOTE: A 37-mm, cellulose ester membrane filter in a cassette preceding the Hydrar may be used if particulate mercury isto be determined separately.
2. Personal sampling pump, 0.15 to 0.25 L/min, with flexible connecting tubing.
3. Atomic absorption spectrophotometer with cold vapor generation system (see Appendix) or cold vapor mercury analysis system.*
4. Strip chart recorder.
5. Flasks, volumetric, 50-mL, and 100-mL.
6. Pipet, 5-mL, 20-mL, others as needed.
7. Micropipet, 10- to 1000-µL.
8. Bottles, biological oxygen demand (BOD), 300-mL.

*See SPECIAL PRECAUTIONS

SPECIAL PRECAUTIONS: Mercury is readily absorbed by inhalation and intact skin. Operate the mercury system in a hood, or bubble vented mercury through a mercury scrubber.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break ends of sampler immediately prior to sampling. Attach sampler to pump with flexible tubing.
3. Sample at an accurately known flow rate of 0.15 to 0.25 L/min for a sample size between 2 and 100 L.
   NOTE: Include a minimum of three unopened sampling tubes from the same lot as the samples for use as media blanks.
4. Cap sampler and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the Hydrar sorbent and the front glass wool plug from each sampler in separate 50-mL volumetric flasks.
6. Add 2.5 mL conc. HNO_3 followed by 2.5 mL conc. HCl.
   NOTE: The mercury must be in the oxidized state to avoid loss. For this reason, the nitric acid must be added first.
7. Allow the sample to stand for 1 hour or until the black Hydrar sorbent is dissolved. The solution will turn dark brown and may contain undissolved material.
8. Carefully dilute to 50 mL with deionized water. (Final solution is blue to blue-green).
9. Using a volumetric pipet, transfer 20 mL of the sample to a BOD bottle containing 80 mL of deionized water. If the amount of mercury in the sample is expected to exceed the standards a smaller aliquot may be taken, and the volume of acid adjusted accordingly. The final volume in the BOD bottle must be 100 mL. To prevent possible loss of mercury during transfer, place the pipet tip below the surface of the liquid in the BOD bottle.

CALIBRATION AND QUALITY CONTROL:

10. Prepare a minimum of two series of working standards covering the range 0.01 to 0.5 µg Hg per aliquot by adding known amounts of the intermediate standard to BOD bottles containing enough 1% nitric acid to bring the final volume to 100 mL.
11. Analyze the working standards together with the samples and blanks (steps 13 through 16). Analyze full set of standards at the beginning of the run, and a second set at the end of the run. Additional standards may be run intermediately during the analysis to confirm instrument response.
12. Prepare calibration graph (peak height from the recorder vs. solution concentration, µg/sample).

MEASUREMENT:

13. Zero the spectrophotometer by removing the bubbler from the BOD bottle, allowing the baseline on the recorder to stabilize.
14. Place the bubbler in a BOD bottle containing 0.5 µg mercury in 100 mL 1% nitric acid. Adjust the spectrophotometer so that it will give a 75% to full-scale deflection of the recorder.
15. Vent the mercury vapor from the system.
16. Analyze standards, samples and blanks (including media blanks).
    a. Remove the bubbler from the BOD bottle.
    b. Rinse the bubbler with deionized water.
    c. Allow the recorder tracing to establish a stable baseline.
    d. Remove the stopper from the BOD bottle containing the next sample to be analyzed. Gently swirl the BOD bottle.
    e. Quickly add 5 mL 10% stannous chloride solution.
    f. Quickly place the bubbler into the BOD bottle.
    g. Allow the spectrophotometer to attain maximum absorbance.
    h. Vent the mercury vapor from the system.
    i. Rinse the bubbler using deionized water.
    i. Place the bubbler into an empty BOD bottle. Continue venting the mercury until a stable baseline is obtained.
    j. Close the mercury vent.

CALCULATIONS:

17. Calculate the amount of mercury in the sample aliquot (W,µg) from the calibration graph.
18. Calculate the concentration C (mg/m^3), of mercury in the air volume sampled, V (L):

$$C = [W \bullet (V_s/V_a) - B]/V$$

Where: Vs = original sample volume (step 8; normally 50 mL)
Va = aliquot volume (step 9; normally 20 mL)
B = average amount of mercury present in the media blanks

EVALUATION OF METHOD:

Rathje and Marcero originally used Hopcalite (MSA, Inc.) as the sorbent material [8]. Later, Hopcalite was shown superior to other methods for the determination of mercury vapor [9]. Atmospheres of mercury vapor for the study were dynamically generated in the range 0.05 to 0.2 mg/m^3 and an adsorbent tube loading of 1 to 7 µg was used. The Hydrar material used in the present method is similar to Hopcalite. No significant difference in the laboratory analysis of mercury collected on the two sorbent materials was observed [10]. OSHA also validated a method for mercury using Hydrar [3]. An average 99% recovery, with s_r = 0.042, was seen for 18 samples with known amounts (0.9 to 3 µg) of mercury added (as $Hg(NO_3)_2$) [11]. No change in recovery was seen for samples stored up to 3 weeks at room temperature or up to 3 months at −15 °C; longer storage times were not investigated [11].

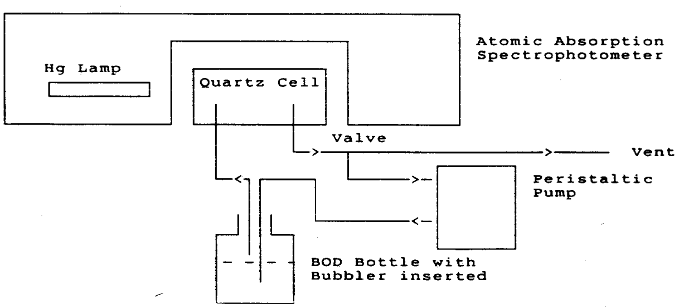
REFERENCES:

[1] NIOSH Testimony, OSHA Proposed Rules on Air Contaminants, Docket #H-020, August 1, 1988.

[2] Evaluation of Mercury Solid Sorbent Passive Dosimeter, Backup Data Report, Inorganic Section, OSHA Analytical Laboratory, Salt Lake City, Utah, 1985.
[3] Mercury in Workplace Atmospheres (Hydrar Tubes). Method ID 145H, Inorganic Section, OSHA Analytical Laboratory, Salt Lake City, Utah, 1987.
[4] NIOSH/MRSB. Reports for Analytical Sequence Nos. 5854, 5900, 6219, and 6311, NIOSH (Unpublished, 1987-1988).
[5] NIOSH Manual of Analytical Methods, 3rd. ed., Method 6000. (1984).
[6] NIOSH Manual of Analytical Methods, 2nd. ed., V.4, S199, U.S. Dept. of Health, Education, and Welfare Publ. (NIOSH) 78-175 (1978).
[7] Ibid., V.5, P&CAM 175, Publ. (NIOSH) 79-141 (1979).
[8] Rathje, A. O., Marcero, D. H. Improved hopcalite procedure for the determination of mercury in air by flameless atomic absorption, Am. Ind. Hyg. Assoc. J. 37:311-314 (1976).
[9] McCammon, C. S., Edwards, S. L., Hull, R. D., Woodfin, W. J., A comparison of four personal sampling methods for the determination of mercury vapor, Am. Ind. Hyg. Assoc. J., 41:528-531 (1980).
[10] Internal Methods Development Research, Data Chem, Inc., Salt Lake City, Utah (1982).
[11] Eller, P.M., NIOSH, unpublished data (1987-88).

METHOD WRITTEN BY: Keith R. Nicholson and Michael R. Steele, Data Chem, Inc., Salt Lake City, Utah. under NIOSH contract No. 200-87-2533.

APPENDIX: Cold Vapor Mercury Analysis System



1. The valve should direct the vented vapors to a hood or to a mercury scrubber system.
2. When the valve is opened to "Vent" the peristaltic pump should draw room air. Place a Hydrar tube in the air intake to eliminate any mercury that may be present.
3. Adjust the peristaltic pump to a flow which will create a steady stream of bubbles in the BOD bottle, but not so great that solution droplets enter the tubing to the quartz cell.
4. If water vapor condenses in the quartz cell, heat the cell slightly above room temperature by wrapping it with a heating coil and attaching a variable transformer.
5. The bubbler consists of a glass tube with a bulb at the bottom, slightly above the bottom of the BOD bottle. The bulb contains several perforations to allow air to escape into the solution (in a stream of small bubbles). A second tube is provided to allow the exit of the vapor. The open end of the second tube is well above the surface of the liquid in the bottle. The two tubes are fixed into a stoppering device (preferably ground glass) which fits into the top of the bottle. A coarse glass frit can be used in place of the bulb on the first tube. However, it is more difficult to prevent contamination when a frit is used.
6. Replace the flexible tubing (Tygon or equivalent) used to connect the bubbler, cell, and pump periodically to prevent contamination due to adsorption of mercury.